Flexural strength, fracture toughness, and translucency of cubic/tetragonal zirconia materials

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Abstract: STATEMENT OF PROBLEM The development of zirconia materials with optimized properties has been rapid, and studies comparing the mechanical and optical properties of recently introduced zirconia with lithium disilicate materials are limited. PURPOSE The purpose of this in vitro study was to compare the mechanical and optical properties of cubic/tetragonal zirconia materials with those of a lithium disilicate ceramic. MATERIAL AND METHODS Specimens were fabricated from 6 different noncolored zirconia materials: Ceramill Zolid FX (CZ), CopraSmile (CS), DD cubeX^2 (DD), NOVAZIR MaxT (NZ), priti multidisc ZrO (PD), and StarCeram Z-Smile (SC), and 1 lithium disilicate ceramic as a control, IPS e.max Press LT A2 (CG). Four-point flexural strength (N=105/n=15) and fracture toughness using the single-edge V-notched beam (N=105/n=15) were examined according to International Organization for Standardization standard 6872:2015. Translucency (N=70/n=10) was evaluated with an ultraviolet spectrophotometer. Grain size (N=6/n=1) of zirconia was investigated by using scanning electron microscopy. Data were analyzed using the Kolmogorov-Smirnov test, multivariate analysis, 1-way analysis of variance, followed by the post hoc Scheffé test and Kruskal-Wallis and Mann-Whitney U tests, and Weibull analysis, using the maximum likelihood estimation method at 95% confidence level (=.05). RESULTS Zirconia materials showed higher mechanical and lower optical properties than CG (P<.001). No differences were observed among the zirconia materials with respect to flexural strength (P=.259) or fracture toughness (P=.408). CG and CS showed significantly higher Weibull modulus than SC and PD. The lowest translucency values were measured for NZ and SC, followed by CS, DD, and PD (P<.001). CZ showed the highest translucency values (P<.001). The lowest grain sizes were found for NZ, DD, and SC; the largest were shown for CS (P<.001). CONCLUSIONS Cubic/tetragonal zirconia showed better mechanical properties than lithium disilicate ceramic. However, the optical properties and the reliability of zirconia are lower than those of lithium disilicate ceramic.

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Flexural strength, fracture toughness, and translucency of new cubic/tetragonal zirconia materials

Short title: Properties of cubic/tetragonal zirconia

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ABSTRACT

Statement of problem. The development of zirconia materials with optimized properties is fast and studies comparing the mechanical and optical properties of esthetic new generation zirconia and lithium disilicate are very limited. Thus, regular scientific updates and expertise is needed.

Purpose. To investigate the mechanical and optical properties of cubic/tetragonal zirconia materials compared with a lithium disilicate ceramic.

Material and methods. Specimens were fabricated from 6 different non-colored zirconia materials: Ceramill Zolid FX (CZ), CopraSmile (CS), DD cubeX² (DD), NOVAZIR MaxT (NZ), priti multidisc ZrO_2 (PD), and StarCeram Z-Smile (SC), and lithium disilicate ceramic as control group: IPS e.max Press LT A2 (CG). 4-point flexural strength (N=105/n=15) and fracture toughness using single edge V-notched beam (SEVNB) (N=105/n=15) were examined according to ISO 6872:2015. Translucency (N=70/n=10) was evaluated with an UV/Vis spectrophotometer. Grain size (N=6/n=1) of zirconia was investigated with scanning electron microscope (SEM). Data were analyzed using Kolmogorov-Smirnov test, multivariate analysis, 1-way ANOVA, followed by post-hoc Scheffé test, Kruskal-Wallis, Mann Whitney U tests, and Weibull analysis using the maximum likelihood estimation method at 95% confidence level (P<.05).

Results. Zirconia materials showed higher mechanical and lower optical properties than CG (P<.001). No differences between all tested zirconia materials were observed with respect to flexural strength (P=.259) and fracture toughness (P=.408). CG and CS showed significantly higher Weibull modulus than SC and PD. The lowest translucency values were measured for NZ and SC, followed by CS, DD, and PD (P<.001). CZ showed the highest translucency values (P<.001). The lowest grain sizes showed NZ, DD, and SC, the largest CS (P<.001).

Conclusions. Cubic/tetragonal zirconia showed higher mechanical properties than lithium disilicate ceramic. However, the optical properties and the reliability of zirconia is lower than those of lithium disilicate ceramic.

CLINICAL IMPLICATIONS

Cubic/tetragonal zirconia and lithium disilicate ceramic are characterized by specific advantages for certain indications. With sole regard to the different generations of zirconia materials, the operator should be aware of relevant differences in mechanical and optical properties.

INTRODUCTION

Nowadays the patients' demand constantly increases when it comes to dental esthetics. In this context, tooth-colored and metal-free monolithic (full contour) restorations specifically made of zirconia are attracting attention. Besides the esthetic aspect, this is due to its immense level of biocompatibility[1] as well as its excellent mechanical properties.[2-4] Highly-stable monolithic zirconia has been typically used in the posterior region (second generation), whereas for the anterior teeth mainly monolithic lithium disilicate has been favored. This may change with the recent introduction of esthetic, highly translucent cubic/tetragonal zirconia (third generation) and ideally result in a simple and efficient manufacturing process, together with increased stability toward lithium disilicate ceramic.[5,6,7,8] Zirconia is a polymorphous material which can form 3 crystal lattices such as monoclinic (room temperature up to 1170°C), tetragonal (between 1170°C and 2360°C), and cubic (about 2360°C to the melting point of 2680°C).[9] Without the addition of stabilizing oxides, such as MgO, CeO_2, or Y_2O_3, a transformation expansion of approximately 3 to 4% from the tetragonal to monoclinic phase takes place followed by cracking.[1] The first generation of zirconia (3Y-TZP) is a partially stabilized tetragonal zirconia (PSZ), with low translucency, an opaque character, and high flexural strength.[3,10] This generation showed translucency only on human dentin grade and hence was not a good fit for monolithic tooth restorations from the esthetic point of view. Thus, it was being applied as a framework material veneered with esthetic ceramics.[11] To avoid high costs, chipping problems, and exceeding reduction of natural tooth, monolithic zirconia restorations without veneering are manufactured.[5,6] Therefore, improvements of the optical properties are still required. Research and development were conducted to enhance translucency of the first zirconia generation. Not only increasing the end temperature of sintering led to microstructural alteration and higher translucency.[12] The duration of holding time, rise in temperature, and cooling while sintering also affected the translucency.[13] Sintering temperatures from 1600°C lead to a decrease of flexural strength[12], which is probably the reason why the first monolithic zirconia generation failed to get accepted. The second generation of zirconia was developed by reducing the number and grain size of alumina together with repositioning them on the grain boundaries of zirconia. This resulted in a higher transmission of light with steadily good long-term stability and high strength.[10] The second generation is still considered inferior in translucency and thus esthetics compared to lithium disilicate ceramic, which has increased the demand for an even more translucent zirconia. Recently, the highly translucent monolithic zirconia materials of third generation have been presented. This zirconia is a fully stabilized cubic/tetragonal zirconia (FSZ). The amount of approximately 50% of the cubic phase has been achieved with higher addition of stabilizing oxides, which resulted in higher translucency due to the more voluminous and more isotropic cubic crystals[13,15] and high strength of the material.[16] It can be reasonably assumed that with the introduction of new generation zirconia, now zirconia materials can both be used for posterior tooth restorations and for esthetic anterior ones. Since very limited research is existing of new zirconia so far, the purpose of this study was to investigate the properties of this zirconia in comparison to a glass ceramic. The null hypotheses stated that no disparities concerning flexural strength, fracture toughness, and translucency are found among 6 cubic/tetragonal zirconia materials and the lithium disilicate ceramic.

MATERIAL AND METHODS

Six cubic/tetragonal zirconia materials were analyzed concerning flexural strength, fracture toughness, translucency, and grain size: Ceramill Zolid FX, CopraSmile, DD cubeX², NOVAZIR MaxT, priti multidisc ZrO_2, and StarCeram Z-Smile. Lithium disilicate ceramic (IPS e.max Press) was used as control group (Table 1).

In summary, 280 specimens were produced. Each material contained 15 specimens for flexural strength, 15 for fracture toughness, and 10 for translucency including 3 for grain size analysis. Specimens were milled from partially-sintered zirconia blanks using a CAM machine (Ceramill motion 2; AmannGirrbach AG) (Fig. 1. A, B). After grinding using SiC abrasive papers P1200 and P2500 (Buehler) specimens were sintered (LHT 02/16; Nabertherm GmbH) pursuant to the directions of the manufacturers (Table 2). For flexural strength and fracture toughness, the final dimension of specimens was 45.0 mm (length) × 4.0 ±0.2 mm (width) × 3.0 ±0.2 mm (thickness) and 16.0 mm (diameter) × 1.0 ±0.05 mm (thickness) for translucency specimens. The specimen production of the lithium disilicate ceramic was carried out by press technique[17] (Austromat 654 press-i-dent; Dekema Dental-Keramiköfen) with 3 wax specimens in each muffle at 930°C. After deflasking and severing, the specimens were polished using a water-cooled polishing machine (Abramin; Struers) with a polishing plate. For flexural strength and fracture toughness, the final dimension of the specimens was 30.0 mm (length) × 4.0 ±0.2 mm (width) × 3.0 ±0.2 mm (thickness) and 16.0 mm (diameter) × 1.0 ±0.05 mm (thickness) for translucency specimens. For translucency, specimens were polished using diamond pads (40 µm and 20 µm), magnetic supporting grinding discs (9 µm and 3 µm), plus a polishing pad (1 µm) with the aid of diamond suspensions (Struers). For lithium disilicate flexural strength and fracture toughness, specimens were polished using SiC paper P500, P1200, P2000, and P4000 (Struers) for wet grinding of the ceramic. Ahead of all tests, the metes of the specimens were measured at 3 points (top end, middle, and lower end of bars) to the precision of .01 mm via a digital micrometer screw (Mitutoyo). Besides, parallelism was verified.

Four-point flexural strength was measured according to ISO 6872:2015. The wide side of the specimen (4.0 ±0.2 mm) was placed in the adapted specimen holder onto 2 steel rolls at distance of 40 mm to each other. Specimen was exposed to loading in a universal testing machine (1445 Zwick/Roell; Zwick) at a crosshead speed of 1 mm/min until fracture. Force was exerted by the plunger apparatus with 2 steel rolls at distance of 20 mm to each other. For smaller lithium disilicate specimens, an adapted specimen holder with the distance of 10 mm between the 2 steel rolls as well as a plunger apparatus with 2 steel rolls at distance of 5 mm to each other was used. Following equation was used for calculating the flexural strength: $\sigma = \frac{3Fd}{4bh^2}$ (σ: flexural strength (MPa), F: fracture load (N), d: difference in the distance of steel rolls (for specimen holder and plunger apparatus respectively) (mm), b: width of specimen (mm), h: height of specimen (mm)).

Fracture toughness was measured according to ISO 6872:2015 (SEVNB method[18]). Five specimens were placed on the narrow side (3.0 ±0.2 mm) and fixed upright, side by side and centered in an adapted specimen holder. A saw cut was inserted in center using a universal cutting machine (Secotom-50; Struers) with a diamond charged cut-off wheel (127 mm dia. × 0.4 mm, Diamond cut-off wheel M1D13; Struers) (Fig. 1. C). The depths of the saw cuts were more than 0.5 mm according to the standard. After that, the specimen holder was placed in a specially constructed notching machine (SD Mechatronik). Here, specimens were notched and sharpened using a razor blade (0.3 mm blades; David combi & finisher) with polishing diamond paste of 9 µm and 3 µm (MetaDi diamond paste; Buehler) (Fig. 1. D). Following the standard, the depth of the saw cut together with the depth of the notching was in between 0.8 mm and 1.2 mm. The cycles of the movement of the machine varied as well as the pressing force of the movement via weights. Specimens were ultrasonically cleaned (Sonorex RK102H; Bandelin electronic) in 80% alcohol (Alkopharm 80; Brüggemann Alcohol) and a steam jet. The saws and notches were measured using a microscope (Zwick/Roell Z 2.5; Zwick) (Fig. 1. E). Specimens were measured with the digital micrometer screw and singly placed into the same adapted specimen holder as for 4-point flexural strength, but this time lying on the narrow side (3.0 ±0.2 mm) with the notched surface pointing downwards. Specimens were exposed to loading in the universal testing machine (1445 Zwick/Roell) at a crosshead speed of 0.5 mm/min until fracture (Fig. 1. F). Following equation was used for calculating the fracture toughness: $K_{Ic} = \frac{F}{b\sqrt{w}} \cdot \frac{s_1 - s_2}{w} \cdot \frac{3\sqrt{a}}{2(1-\alpha)^{1,5}} Y$ (K_{Ic}: fracture toughness (MPa√m), F: fracture load (N), b: thickness of specimen (m), w: width of specimen (m), s_1: bearing range (m), s_2: inner range (m), α: relative depth of the V-notch, Y: form factor of stress intensity)

Translucency was analyzed with a UV/Vis spectrophotometer (Lambda 35; PerkinElmer LAS). All specimens were cleansed with 80% alcohol and singly placed in the appropriate specimen holder with the aid of barium sulfate modelling mass. Specimens were loaded in the spectrophotometer at the inlet hole of the integrating sphere. Quantitative measurements of translucency were implemented by analyzing the definite transmission of light through each specimen. The spectrophotometer, using a dual beam system, records the light transmission with the help of a sensor in comparison to the light intensity from a split beam.

The light source provided a wavelength varying between 400 and 700 nm. Initial translucency was calculated by the intensity of the monochromatic light I_0, and the light I, transmitted through the specimen. The transmission coefficient t_c [%] was calculated using following equation: $I/I_0 = t_c^x$. The overall light transmission for each specimen [T] was calculated as the integration (t_c (λ) dλ [10^{-5}]) of all t_c values for the wavelengths from 400 to 700 nm. To analyze the light transmission, the T value of each material was divided by the T value with no specimen in the spectrophotometer (baseline), to receive light transmission in %. All tests were conducted at room temperature.

Grain size analysis of zirconia was conducted by scanning electron microscopy (SEM, Carl Zeiss Supra V50, Cathode: field emission; Carl Zeiss Microscopy GmbH). The preparation involved thermal etching in the furnace (LTH 02/16; Nabertherm) with 1450°C final temperature and 30 minutes holding time. Subsequently, specimens were cleansed with 80% alcohol and singly glued on the appropriate holder. Each specimen was sputtered with gold for 45 s (layer thickness: 2 nm, Sputter Coater, Safematic CCU-010; Safematic). SEM was operated with an acceleration voltage of 5 kV at a working distance of 8.4 to 10.0 mm. Grain sizes were determined at 3 different spots on the same specimen.

The measured data were analyzed statistically with SPSS Version 24.0 (IBM, SPSS, Statistics). Parametric and non-parametric descriptive statistics were computed. For quantitative variables, the assumption of normality was tested with the Kolmogorov-Smirnov test. The general linear model (multivariate) analysis was performed. Flexural strength and grain size data were analyzed using one-way ANOVA followed by post-hoc Scheffé test. Translucency and fracture toughness were tested with non-parametric tests, such as Kruskal-Wallis and Mann Whitney U tests. Statistical analyses were performed for all materials, while zirconia materials were also considered separately. Weibull distribution parameters (Weibull modulus, characteristic strength) for flexural strength values were calculated using the maximum likelihood estimation method at 95% confidence level.[18] The results of statistical analyses with $P<.05$ were interpreted as statistically significant.

RESULTS

According to the multifactorial analyses, the tested materials showed an impact on all parameters, namely flexural strength, fracture toughness, translucency, and grain size. The highest influence on the ceramic materials showed the translucency (partial eta squared η_P^2 = 0.979, $P<.001$), followed by the flexural strength (η_P^2 = 0.524, $P<.001$), the fracture toughness (η_P^2 = 0.430, $P<.001$), and the grain size (η_P^2 = 0.383, $P<.001$).

The Kolmogorov-Smirnov test indicated higher rate of violations of the normality assumption for translucency (28%) and fracture toughness data (14%) (Table 3). The violation of the normality assumption was not caused by outliers but rather by measurement rounding, leading to an increased coarseness of the observations in each test group. Consequently, translucency and fracture toughness data were analyzed non-parametrically. Within flexural strength and grain size data, all groups were normally distributed and analyzed using parametric tests (Table 3).

The control group, IPS e.max Press showed the lowest flexural strength ($P<.001$) and fracture toughness ($P<.001$) but the highest translucency ($P<.001$) compared to tested zirconia materials. No statistical differences between all tested zirconia materials were observed with respect to flexural strength ($P=.259$) and fracture toughness ($P=.408$). IPS e.max Press (m=8.8) and CopraSmile (m=8.4) showed significantly higher Weibull modulus than StarCeram Z-Smile (m=4.9) and priti multidisc ZrO_2 (m=4.4) (Table 4). Within the zirconia materials, CopraSmile (m=8.4) showed a significantly higher Weibull Modulus compared to priti multidisc ZrO_2 (m=4.4) and StarCeram Z-Smile (m=4.9).

With respect to translucency, the lowest values were measured for NOVAZIR MaxT and StarCeram Z-Smile, followed by CopraSmile, DD cubeX^2, and priti multidisc ZrO_2 (*P*<.001). Ceramill Zolid FX showed the highest translucency values (*P*<.001).

The lowest mean grain size was determined for NOVAZIR MaxT, DD cubeX^2, and StarCeram Z-Smile (*P*<.001) (Table 3). The significantly largest value was measured for CopraSmile (*P*<.001).

DISCUSSION

This study tested the mechanical and optical properties of cubic/tetragonal zirconia in comparison to a lithium disilicate ceramic. The hypothesis, that flexural strength, fracture toughness, and translucency of tested zirconia are comparable to lithium disilicate ceramic is rejected. The flexural strength values varied between 490 MPa (DD cubeX^2) and 557 MPa (Ceramill Zolid) depending on the zirconia material. Comparable results of flexural strength were found in current literature that investigated the impact of different zirconia compositions on the flexural strength.[16] The lithium disilicate ceramic showed lower values (296 MPa). The flexural strength data were analyzed with Weibull distribution, predicting failure chance at any level of stress. With the help of the Weibull results, the reliability of the materials can be compared. The Weibull modulus of zirconia materials varied greatly. This observation might be attributed to different sintering parameters of the respective manufacturers. CopraSmile showed the highest Weibull modulus and was sintered at 1500°C final temperature, while the remaining zirconia materials, sintered at 1450°C, resulted in lower Weibull modulus. In accordance to the higher sintering temperature of 1500°C, CopraSmile showed the largest mean grain size. Previous studies of first generation zirconia observed a correlation between grain size and translucency as well as flexural strength results.[12] CopraSmile showed a comparable Weibull modulus to the lithium disilicate ceramic.

Compared to other studies, almost twice as high Weibull modulus values were achieved for the control group which might be caused by the measuring method.[17] In the previous study, the 3-point flexural strength was measured while the present measurements focused on 4-point flexural strength. With respect to the conducted 4-point bending test it is to remark that the distance between the steel rolls, that transferred the load onto the specimens, differed according to the dimension of specimens. For lithium disilicate ceramic specimens, a smaller distance between the steel rolls of the plunger apparatus was used. This might have an impact on the outcoming values because of differences in the stress distribution and result in overestimated values of flexural strength for the lithium disilicate ceramic. This limitation should be considered and tested using an appropriate test method in further investigations. For fracture toughness, the 4-point single edge V-notched beam (SEVNB) method was applied. It must be pointed out, that the standards demand for a certain sharp notch root radius. This was extremely difficult to implement for the fine-grained microstructure of the zirconia materials used in this study.[14] Previous studies stated that the results for fracture toughness measured by SEVNB would be overestimated, if the notch root radius is above a critical value of about 1.5 to 3 times the mean grain size.[14,19] The mean grain size of zirconia was between 594 nm and 903 nm. Thus, a maximum notch root radius between 1 to 2 µm is needed to measure the true fracture toughness. This condition was not satisfied for the zirconia materials used in this study. Thus, the fracture toughness values might be slightly overestimated, but still the comparability among tested materials is given. Corresponding results were shown in a previous study.[14] Here, the mean grain size of 5 different zirconia materials varied between 250 nm and 700 nm, which involve a maximum root radius between 1 to 2 µm, while the notch root radius was about 5 to10 µm. In this study, the zirconia materials resulted in significantly higher fracture toughness than IPS e.max Press. All zirconia materials lied within the same value range (3.34 to 3.77 MPa√m) while IPS e.max Press showed values of 2.10 MPa√m.

Lithium disilicate ceramic presented higher translucency than zirconia. Corresponding results were shown in a previous study.[15] Here, IPS e.max CAD LT showed higher translucency than different zirconia materials for monolithic restorations. In the present study, statistical differences between all tested zirconia materials were found whereat Ceramill Zolid demonstrated the highest translucency. According to the manufacturers recommendations, the investigated materials were sintered with different parameters in heating rate, holding time, and cooling rate (Table 2). Therefore, an impact of the sintering parameters on the translucency of the materials is suspected. Regarding to the conducted measurements of translucency, it must be emphasized, that all specimens provided the same thicknesses of 1.0 ±0.05 mm. But since lithium disilicate restorations at least require 1.5 mm to 2.0 mm of occlusal thickness to withstand mechanical stress in the oral cavity, cubic/tetragonal zirconia may be successfully used for monolithic tooth restorations with reduced occlusal thickness and therefore less occlusal tooth reduction.[7,8,15] Further clinical studies are demanded to confirm that.

Returning to the impact of sintering parameters, various investigations demonstrated, that the time and temperature effect the translucency, grain size, eventual defects, flexural strength, and fracture toughness. The present study observed remarkable diversify in grain sizes, although end temperature and sintering time accumulate virtually to similar energy input for the zirconia materials except for CopraSmile. Last-mentioned demonstrated the impact, since it has evolved the largest grain sizes and was sintered at the highest temperature of 1 500°C and presumably with a long cooling time because of the undefined cooling process recommended by the manufacturer. For the other materials, such coherences were not present.

According to the studies available to date, it was proved that higher addition of stabilizing oxides implemented approximately 50% of the cubic phase in third generation zirconia.[14] This gave rise to translucency, even though the flexural strength and fracture toughness were sacrificed. Current literature also proved that the composition of zirconia materials influences flexural strength and translucency values. It was stated that the addition of varying amounts of Y_2O_3 and Al_2O_3 resulted in new materials with intermediate properties in between the conventional zirconia and lithium disilicate ceramic.[16] The present results are in accordance with this statement.

One limitation of this study was the lack of power analysis conducted at its planning stage. When choosing 15 observations for fracture toughness and flexural strength and we relied to some extent on the experience from a previous study based also on 15 observations.[10] For contrast ratio/translucency 10 specimens were choice and for grain size determination 3 specimens.[12,13]

However, clinical situations can only be estimated by laboratory tests. Therefore, further clinical studies are necessary to assess the optical and mechanical properties of new zirconia and lithium disilicate ceramics. In addition, thermodynamic stability of these zirconia materials has not yet been adequately tested and should be thoroughly investigated in the following studies. In general, knowledge about the third generation of zirconia is not sufficient and needs to be further researched.

CONCLUSION

Within the limitations of the study, following conclusions can be drawn:

In terms of mechanical properties, the third generation zirconia can replace the lithium disilicate ceramic in some clinical applications. However, the esthetic properties are still better for glass ceramics than for oxide ceramics. Also in terms of reliability, only one zirconia (CopraSmile) could reach the values of the lithium disilicate ceramics.

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TABLES

Table 1. Summary of used materials with lot numbers.

Material Group	Material	Abbreviation	Manufacturer	Lot Number
Zirconia third generation	Ceramill Zolid FX	CZ	Amann Girrbach, Koblach, Austria	1512004-114
	CopraSmile	CS	Whitepeaks Dental Solutions, Wesel, Germany	IS2114
	DD cubeX^2	DD	Dental Direkt, Spenge, Germany	8041632001
	NOVAZIR MaxT	NZ	Novadent Dentaltechnik, Hamburg/Dresden, Germany	L2251030007-6
	priti multidisc ZrO_2	PD	Pritidenta, Leinfelden-Echterdingen, Germany	Y65 16HT
	StarCeram Z-Smile	SC	H.C.Starck, Munich, Germany	50586461
Lithium disilicate (CG)	IPS e.max Press LT (A2)	CG	Ivoclar Vivadent, Schaan, Liechtenstein	U04298

Table 2. Sintering parameters used for tested zirconia materials.

Groups	Heating rate from room temperature and eventual heating steps	Final temperature (°C)	Holding time (min)	Cooling rate up to 25°C (°C/min)
CZ	8°C/min	1450	120	15
CS	10°C/min until 950°C are attained, then 6°C/min	1500	90	unregulated in closed furnace
DD	8°C/min until 900°C are attained; after holding for 30min, heating rate of 3°C/min	1450	120	10
NZ	4°C/min until 500°C are attained, then 8°C/min until 1150°C are attained; after holding for 30min, 2°C/min to 1300°C, then 4°C/min	1450	120	8 to 800°C, then 0 to 100°C
PD	10°C/min	1450	120	10
SC	5°C/min until 900°C are attained; after holding for 30min, 2.5°C/min	1450	120	5 to 900°C, then unregulated

Table 3. Parametric and non-parametric descriptive statistics for all generated data.

		Parametric analysis		Non-parametric analysis		
Material group	material	Mean ± SD	95% CI	Min	Median	max
Flexural strength [MPa]						
Zirconia	CZ	557 ± 88^{b}	507;606	361	564	673
	CS	507 ± 69^{b}	467;545	383	508	630
	DD	490 ± 83^{b}	443;536	357	495	654
	NZ	540 ± 86^{b}	491;588	317	551	658
	PD	493 ± 119^{b}	425;559	208	481	731
	SC	498 ± 104^{b}	439;556	260	504	629
LiSi (CG)	CG	296 ± 39^{a}	274;318	229	294	361
Fracture toughness [MPa*m$^{1/2}$]						
Zirconia	CZ	3.56 ± 0.47^{b}	3.1;3.9	3.00	3.38	4.50
	CS	3.34 ± 0.56*^{b}	2.9;3.7	2.25	3.32	4.62
	DD	3.64 ± 0.71^{b}	3.1;4.1	2.87	3.52	5.39
	NZ	3.69 ± 0.88^{b}	3.1;4.2	2.64	3.41	5.31
	PD	3.34 ± 0.72*^{b}	2.8;3.8	2.73	3.10	5.01
	SC	3.77 ± 0.72^{b}	3.2;4.2	2.86	3.57	5.11
LiSi (CG)	CG	2.10 ± 0.14^{a}	1.9;2.2	1.85	2.09	2.47
Translucency [%]						
Zirconia	CZ	38.3 ± 0.3^{b}	37;39	37.8	38.3	38.7
	CS	37.1 ± 0.3^{c}	35;38	36.4	37.2	37.5
	DD	37.3 ± 0.3^{c}	36;38	36.7	37.3	37.6
	NZ	33.1 ± 0.5^{d}	31;34	32.3	33.3	33.5
	PD	37.6 ± 0.5*^{c}	36;38	36.3	37.6	38.2
	SC	33.6 ± 0.2^{d}	32;34	33.4	33.6	34.0
LiSi (CG)	CG	40.4 ± 0.4^{a}	39;41	39.9	40.5	41.0
Grain size [µm^{2}]						
Zirconia	CZ	0.515 ± 0.49^{bc}	0.393;0.637	0.460	0.534	0.552
	CS	0.817 ± 0.048^{d}	0.696;0.936	0.789	0.789	0.872
	DD	0.373 ± 0.038^{ab}	0.278;0.468	0.331	0.385	0.404
	NZ	0.353 ± 0.015^{a}	0.314;0.390	0.338	0.352	0.368
	PD	0.580 ± 0.041^{c}	0.476;0.683	0.534	0.592	0.614
	SC	0.462 ± 0.063^{abc}	0.304;0.618	0.394	0.473	0.518

*not normally distributed, ^{abc} different letters represent significant differences between materials within one test parameter.

Table 4. Weibull statistics for flexural strength values.

Material group	material	Characteristic strength (∂) [MPa]	95% CI	Weibull modulus (m)	95% CI
Zirconia	CZ	594	548;644	6.9	3.9;11.8
	CS	536	501;572	8.4	4.8;11.4
	DD	523	481;567	6.9	3.9;11.7
	NZ	577	530;626	6.7	3.8;11.4
	PD	538	474;610	4.4	2.5;7.6
	SC	542	483;607	4.9	2.7;8.3
LiSi (CG)	CG	313	292;332	8.8	5.0;15.0

FIGURES

Fig. 1. Fracture toughness specimen preparation step by step. A, Representative image of zirconia blank placed in CAM machine (Ceramill motion 2, Amann Girrbach). B, Representative image of milled specimens for 4-point flexural strength and 4-point fracture toughness (45.0 × 4.0 × 3.0 mm). C, Representative image of saw cutting of specimens (Secotom-50 – Struers). D, Representative image of notching of specimens in notching machine (SD Mechatronik). E, Representative image of microscopy of notched zirconia specimen using SEVNB (magnification 5×). F, Representative image of specimen loaded in universal testing machine (1445 Zwick/Roell).

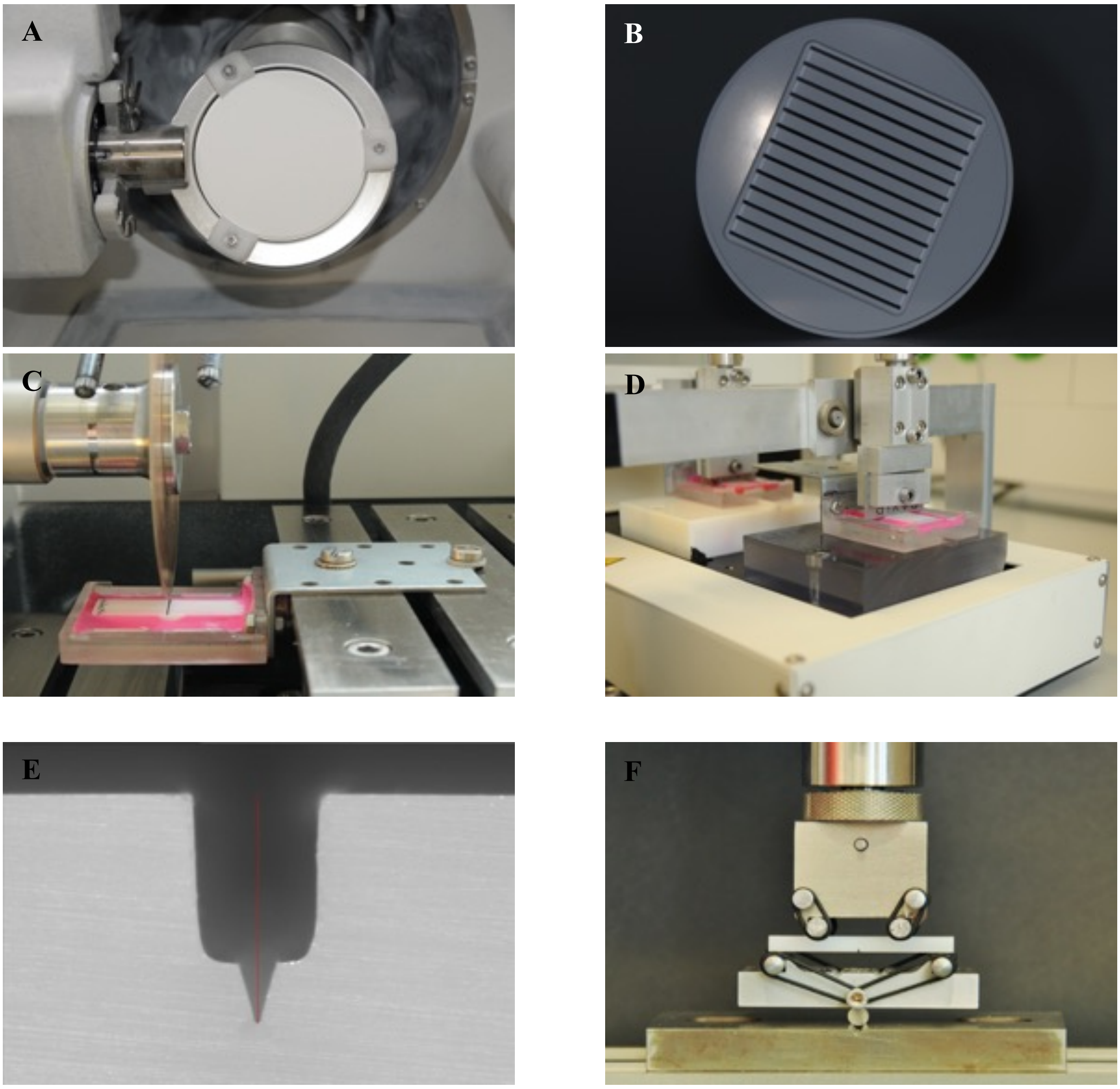

Fig. 2. Representative image of REM micrographs showing grain sizes of polished zirconia plates for translucency (magnification 50.000×).